ULTRASONIC-ASSISTED ADSORPTION OF SUGARCANE PHENOLICS ONTO AGRICULTURAL BYPRODUCT BASED NOVEL BIOSORBENTS

Synopsis
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ORIGIN OF PROBLEM

Carbons were the first materials to be applied to adsorption process (e.g. the Phoenicians first recorded the use of charcoal to purify water in 460 BC). The sugar industry in England (towards the end of the 18th century) is credited with the first use in an industrial process of charcoal and carbons of animal origin (i.e. for decolourizing sugar syrups) [1]. It wasn't until 1941 that Hertzog and Broderick [2] described the production of activated carbon from coal mine waste and its use in sugar decolourizing.

In the last twenty years many new adsorptive materials have been developed. These materials include activated carbon fibers and molecular sieves, microporous glasses and nano-porous materials made from carbonaceous and inorganic materials. The concept of pore size engineering has also been considerably advanced. Pore size engineering can produce adsorbents of essentially identical composition (e.g. activated carbons) with tailored pore size and surface area. By such an approach activated carbons that approach the theoretically possible limit of surface area [3] (ca. 4000 m^2/g) have been produced. *Now carbon adsorption is customary in food processing industries, such as sugar refining and for pollution control as in waste water treatment. Ultrasonic is an effective purification and separation technique used in industry especially in wastewater treatment.*

Thus, the present piece of work is aimed to remove sugarcane phenolics (sugarcane phenolic acid) from aqueous media by green routes using low-cost agricultural based activated adsorbent materials developed from agricultural by-product-based waste materials (***Sugarcane Bagasse Ash, Rice Husk Ash and Corn Cob Ash***) in the presence and absence of Ultrasound (US).

INTRODUCTION

Food processing, sugar refining industries and pollution control agencies are utilizing the adsorption studies extensively, for waste research and bioremediation. The extensive use of activated carbon is generally attributed to its large surface area making it a powerful adsorbent. Granular activated carbon (GAC) is an excellent adsorbent for the removal of dyes, organics, and metals from wastewaters. However, GAC is expensive and the regeneration and reuse of the spent GAC makes it even more expensive. Activated carbon prepared from agricultural by-products (lignocellulosic material) is one of

the potential alternatives for commercially available activated carbon currently used in raw sugar decolorization [4].

The utilization of industrial and agricultural waste produced by industrial processes has been the focus of waste reduction research for economic, environmental, and technical reasons [5-6]. Moreover, using plant parts as adsorbent material is of great interest to many researchers because they have the capacity of adsorbing mineral salts and metal ions onto their surfaces and hence, they serve as "Green Adsorbents". In recent years, there has been a growing research activity on using agricultural/natural wastes as efficient biosorbents. These materials are locally available, low cost, and environmental friendly. These factors have prompted the increased use of such materials as biosorbents. The proposed investigation relates to the removal of sugarcane phenolics (sugarcane phenolic acid) from highly concentrated sugar solutions employing a new kind of adsorbent which is derived from an inexpensive, abundantly available and eco-friendly source.

The efficiency of any adsorbent material to adsorb desired compounds (colour, ash, colloids) depends on several factors [7-8] such as porosity, surface area, pore size distribution, bulk density, surface chemistry, hardness, pH, particle density, particle size, amount of water-soluble minerals and its total ash content. In case of sugar decolourization; the decolourizing material should possess the pore size distribution favourable for adsorption of a mixture of polydispersed constituents from highly concentrated sugar solutions, including the pH-sensitive sugar colourants; such as phenolics and flavonoids [9].

Phytoremediation

Activated carbon from agricultural by-products has the advantage of offering an effective, low cost replacement for existing bone char or coal-based granular activated carbon (GAC). This is a new approach for the treatment of contaminated water or soil. Phytoremediation means the use of plant-based materials for remediation of wastewater or polluted soil. Scientists and researchers are now focusing on search of plant materials that would efficiently remove contaminants from the wastewater. Phytoremediation is used in adsorption processes for more efficient removal of metal ions and TDS from the wastewater [10]. These materials are locally available, low cost, and are environmental friendly.

Selection of Waste Material

- **Sugarcane Bagasse Ash (SBA)**

Sugarcane bagasse Ash (SBA), a waste material generated in the sugar industry, is being indicated as causative agent of serious environmental pollution, which calls for an urgent need to develop ways of handling the waste. Bagasse ash mainly contains alumina and silica. Sugarcane bagasse ash is an important by-product of the sugarcane industry and most of it is burnt to produce steam and electricity in a co-generation plant at the ethanol factory. The sugarcane bagasse ash is the result of the bagasse combustion which mainly consists of high amount of silica (SiO_2) and unburned carbon. It's utilization as an adsorbent as well as mineral admixture in cement and concrete has also been examined [11].

- **Rice Husk Ash (RHA)**

Rice husk ash are an agricultural waste material, which is easily available, cheap and economically advantageous. Rice husk ash is an undesirable agriculture mass residue. In India, is a by-product of the rice milling industry. It is one of the most important agricultural residues in quantity. It represents about 20% of the whole rice produced, on weight basis of the whole rice [12]. It's chosen because of its granular structure, chemical stability and its local availability at very low cost and there is no need to regenerate them due to their low production costs. The main constituents of rice husk are: 64% volatile matter, 12-16% fixed carbon and 15-20% ash [12]. The rice husk compositions are: 32%, cellulose, 21.44% lignin, 21% hemicelluloses, 8.11% water and 15% mineral ash [13].

- **Corn Cob Ash (CCA)**

Corn cob is a lignocelluloses material composing of cellulose, hemicellulose and lignin (Vaughan et al, 2001) thus by carbonizing the precursor a porous carbon adsorbent would be obtained (Rahman *et al.*, 2005). About 11.5 million tons maize is produced in our country annually. Therefore, the corn cobs are easily available free of cost. Several researchers have reported developing activated carbon from corn cobs, but the surface areas were relatively low and the physical adsorption more energy demanding. Different values of surface areas were reported some of which are a value of 437 m^2/g, 664, 850, and 700 m^2/g, 608 and 897 m^2/g, 1014 and 1063 m^2/g, depending on the carbonization [14].

The surface of the corn-cobs can be modified with metal ions to attract anions (sugar colourants) from aqueous solution [15].

Decolourization by Adsorption Studies

Adsorption at various interfaces (liquid -solid, liquid-gas, solid-gas etc.) has been a serious matter of study among the scientists from the beginning and specially during the last century. Among the physico-chemical processes, adsorption technology is considered to be one the most effective and proven technology having potential application in both water and waste water treatment. Decolourization is a result of two mechanisms- adsorption and ion exchange and is influenced by many factors including sugar colour/sorbent interaction, sorbent surface area, particle size, temperature, pH and contact time [10]. Adsorption studies are based on generally two types of adsorption processes:

- **Physisorption or Physical adsorption** – it arises due to the van der Waals interactions between adsorbate and adsorbent.
- **Chemisorption or Chemical adsorption** – it arises due to chemical interactions between adsorbate and adsorbent.
- **Mechanism of adsorption -** Adsorption is a three-step process. At first the adsorbate diffuses from fluid stream to the external surface of adsorbent. Secondly the adsorbate shifts to the pores of the adsorbent particles. Majority of adsorption occurs in these pores because of their large surface area. Finally, the molecules adhere to the surface area of pores. Adsorption is a surface phenomenon which results out of binding forces between atoms, molecules and ions of adsorbate and the surface of adsorbent.

Advantages of adsorption techniques: Adsorption is more advantageous than other techniques (Dabrowski 2001) due to:

- ✓ Low operation cost
- ✓ High flexibility
- ✓ Simple design and operation
- ✓ Easy automation
- ✓ Lack of sensitivity to toxic pollutants

- ✓ Capability of operation at very low concentration
- ✓ Environmentally friendly
- ✓ Less investment in terms of initial cost

Ultrasonic Purification and Separation Technique

Ultrasonic is an effective purification and separation technique used in industry especially in wastewater treatment. Ultrasonic waves generate high-energy which causes the cavitation. Cavitation is the rapid and repeated formation and resulting implosion of micro bubbles in a liquid resulting in the propagation of microscopic shock waves. Microscopic vapor bubbles are created at site of rarefaction as the liquid fractures or tears because of the negative pressure of the sound wave in the liquid. Finally, the compression part, which follows the rarefied part, collapses the bubble. The cavitational collapse of bubble generates localized hot spots of temperature as high as 5000K and pressure as high as 1000 atmospheres for a life time of a few microseconds thus creating high energy movements of the solvent that resultsin localized high energy shear forces [16-17]. The number of cavitation bubbles collapsing per second may well be in the millions hence their cumulative effect can be significant. Shock waves from cavitation in liquid-solid slurries produce high velocity-inter particle collisions, the impact of which is sufficient to convert the solid molecule into semi solid molecule. Thus, the ultrasonic energy may be effective to remove the sorbed contaminants, like heavy metals, from fine grained soils.

Application of Ultrasonic Technique in the Bioremediation

The mechanism causing sonochemical effects in liquids is the phenomenon of acoustic cavitation. The chemical effects of ultrasound are derived from acoustic cavitation, which is a nonlinear process that serves as a means of concentration of the diffuse energy of sound in liquids. This high-energy microenvironment is induced by the extreme conditions generated during acoustic cavitation leading to the generation of high-energy species (radicals) [18-24]. Cavitation is the formation, growth, and implosive collapse of bubbles in a liquid. Cavitation collapse produces intense local heating, high pressures, enormous heating and cooling rates, and liquid jet streams (~400 km h^{-1}) [25-29].

In aqueous sonochemistry, chemical reactions occur at three regions:

(1) The cavitation bubble: Interior of collapsing bubbles where extreme conditions of temperature and pressure exist transiently, which induce chemical reactions yielding products that are typical of pyrolysis or combustion reactions in the gas phase.

(2) Bubble/solution interface: Interfacial regions between the cavitation bubbles and bulk solution where a high temperature (lower than that in the bubble core) is generated [30].

(3) Bulk solution: Where the free radicals, being produced in the interior of bubble and in the interfacial region, that survive migration from the interface can undergo reactions with solutes present in the bulk solution [31-32].

Advantages of using sonochemical method over other

There are immense advantages of using sonochemical method over other, as under:

1. Sonochemical method is faster, therefore more productive in terms of time and energy consumption.
2. Products synthesized by the sonochemical methods have greater stability.
3. The control of particle size is easier in sonochemical process, which is otherwise not possible by any other technique.
4. Preparation of amorphous products is possible, therefore, enhanced characteristics such as adsorption, catalytic activity etc. may be expected.

PRESENT STATE OF KNOWLEDGE

Overview of India's sugar sector, Sugar industry in India after textile is the second largest agriculture-based industry. First sugar mill in India was established in 1930. Currently about 4 million hectares of land in India is under sugarcane with an average yield of 70 tons per hectare. The geographical distribution of sugarcane production in India is shown in Figure 1.

Sugar industry in India is concentrated in states of Uttar Pradesh, Maharashtra, Tamil Nadu, Karnataka, Andhra Pradesh, Gujarat, Haryana and Punjab. Growth of the sugar industry in India [9] is described below in Table1 [http://www.indiansugar.com] [33].

Table 1: Growth of the sugar industry in India

Particulars	2012-13	2013-14	2014-15	2015-16	2016-17	2017-2018	2018-2019
No. of Factories in operation	507	529	526	509	538	526	497

Cane acreage (Thousands Hectares)	4885	5100	5279	5341	5307	5284	4945
Sugar Production (Lakh Tons)	3424	3538	3544	3456	3668	3369	3036
Molasses Production (Thousands Tons)	10970	11824	11744	10882	12482	10837	9026

The sugar sector is an important pillar of the Indian economy as it provides livelihood to 50 million farmers and to 5 million workers directly employed in sugar factory [34] [http://dfpd.nic.in]. Besides this it creates employment in ancillary services in transportation and trade etc. At present sugar sector's output worth is Rs.80, 000crore.

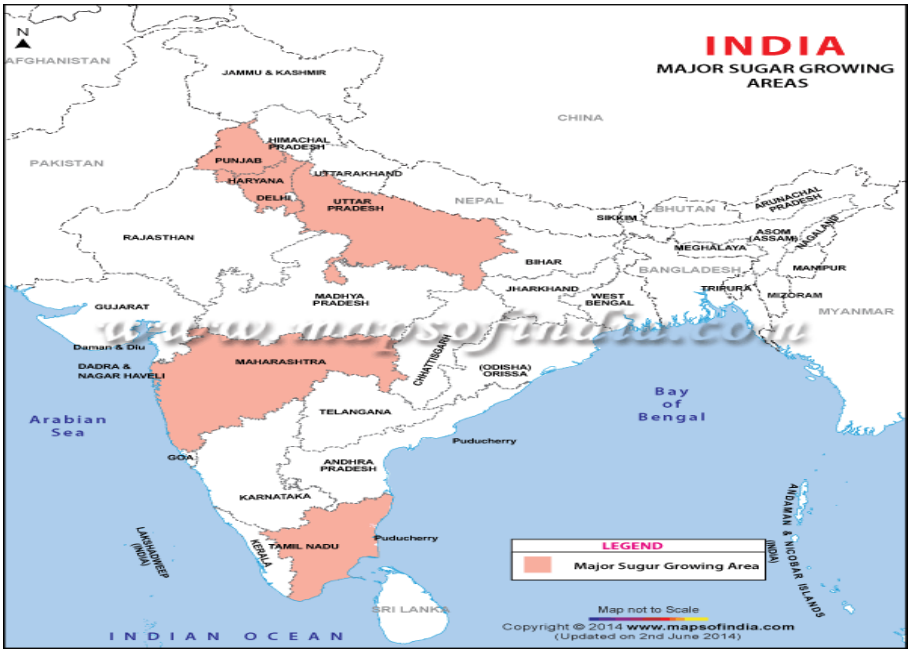


Figure 1: Geographical distribution of sugarcane production in India [33].

The Sugar Mills in India

India produces 18-20 million tons of sugar per year, making it the second largest sugar producer in the world. There are at least 550 sugar mills in India, distributed among 19 states, grinding 1250-2500 tons cane per day [33-34]. The main cane producing states and the number of mills in respective states are mention below:

States	**No. of Mills**
Maharashtra	193
Uttar Pradesh	148
Karnataka	58
Andhra Pradesh	38
Tamil Nadu	48

Gujarat 22

Most sugar plants in India produce the direct consumption sugar known as plantation mill white sugar (PMWS). It is produced from milled cane using double suffixation process.

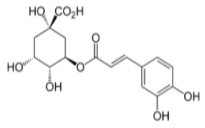
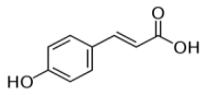
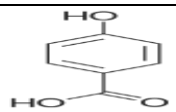
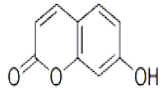
Cane Sugar Colourants

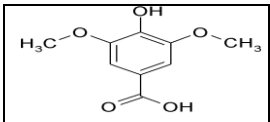
This general term 'colourant' has been coined by Judd (1952) [35] in order to indicate material which is either a dye (a compound which absorbs light with neglible scattering and a pigment (a compound which absorbs light with scattering). The colourants are among the most troublesome impurities in the production of white cane sugar. Despite the diversity of colourants in sugar solutions, presently, colouration is assessed by measuring absorbance at 420 nm as per ICUMSA protocol [35].

Physico-Chemical Properties of the sugarcane phenolics (SCP) or sugarcane phenolic acids (SCPA) selected to study for adsorption

The p- hydroxybenzoic acid, cholorogenic acid, p-coumaric acid, 7-hydroxycoumarin and syringic acid were chosen as representative selection of phenolic acids commonly found sugar cane. The basic information concerning phenolic compounds selected for study are included in Table 2.

Table 2: Physico-Chemical Properties of the sugarcane phenolics (SCP) or sugarcane phenolic acids (SCPA)

Phenolic Compound	Melting Point (°C)	pKa at 25°C	Aqueous Solubility at 25°C (g/100ml)	Density in (g/cm^3) & Surface Tension (dyne/cm)
 Chlorogenic acid	208	pKa= 7.8	Slightly Soluble	1.28 & 101.9
 p- coumaric acid	206	pKa_1= 4.36 pKa_2= 8.98	Slightly soluble	1.33 & 62.42
 p-hydroxybenzoic acid	214	pKa_1= 4.48 pKa_2 = 9.32	0.2	1.46 & 64.41
 7-hydroxycoumarin	226	pKa =9.16	1.0	1.40 & 59.49

 Syringic acid	207	pKa_1= 4.71 pKa_2= 9.12	0.58	1.33 & 51.61

Research Attention

Biosorption is a green approach which uses plant materials as effective adsorbents for treatment of waste water. It is a property of living or dead biomasses to bind and concentrate metal ions from solutions. It is a form of adsorption process involving the removal of ions from liquid phase onto solid biomass material. Some biosorbents are competitive with existing non biomass-based treatments [36]. Most of the plants have the potential to absorb the mineral salts and metal ions through their roots and then store them as organic minerals while some plant parts have the ability to adsorb salts and metal ions onto their surfaces [37]. A lot of research has been done on using plant-based materials as biosorbents; some of the biosorbents and adsorbent materials reported in the literature are presented in the Table 3 below:

Table 3: Biosorbents/Adsorbent materials/Techniques used for sugar decolourization reported in the literature

S. No.	Biosorbents /Adsorbent materials /Techniques	References	References No.
1.	Rice Husk (Activated)	Ahmedna et al., 1997	4
2.	Sugarcane Bagasse Pith	Ahmedna et al., 1997	4
3.	Bagasse Bottom Ash	Simaratanamongkol et al., 2010	38
4.	Defecation, Sulfitation and Carbonation	Michael et al., 2010	39
5.	Functionalized Accurel	Kaman Singh et al., 2011	40
6.	Activated Carbon	Nasehi et al., 2012	41
7.	Modified Sugar Beet Pulp	Hasan et al., 2012	42
8.	Activated Carbon	Zhang et al., 2012	43
9.	Activated Mustard Cake	Kaman Singh et al., 2014	36
10.	Activated Corn Cobs Powder	Ram Bharose et al., 2014	44
11.	Activated Wheat Husk (AWH)	Sudhir Kumar Verma et al., 2014	45
12.	Adsorbent Chromatographic Column	Manarim et al., 2016	46
13.	Powdered Carbon	Hind et al., 2018	47
14.	Activated Charcoal	Shikare Rasika et al., 2018	48
15.	The effect of high intensity ultrasound (HIU) on the kinetics of crystallization of sucrose	Kaman Singh et al., 2019	49

OBJECTIVES

- The proposed work is to examine the removal of sugarcane phenolics (sugarcane phenolic acids) from aqueous media by green routes using low-cost agricultural based activated adsorbent materials developed from agricultural by-product-based waste materials (Sugarcane Bagasse Ash, Rice Husk Ash and Corn Cob Ash) in the presence and absence of ultrasound (US). Raw materials, Sugarcane Bagasse, Rice Husk and Corn Cob are abundantly and cheaply available.
- The effect of operating parameters such as initial concentration of sugarcane phenolic acid, pH, particle size, contact time, temperature, and adsorbent dosage for the removal of sugarcane phenolics (SCP) or sugarcane phenolic acids (SCPA) proposed to be studies in the presence and absence of Ultrasound (US).
- Desorption studies will also be considered to make the proposed green adsorbent economical.
- Adsorption behavior is to be analyzed using Langmuir, Freundlich and Temkin isotherm models.
- The kinetic and the mechanism studies of adsorption will also be examined on the model of Weber-Morris and Boyd.
- The feasibility of sorption process in the environmental conditions, thermodynamics study will be carried out by using Gibbs free energy, Enthalpy and Entropy parameters.

PLAN OF WORK: AT A GLANCE

❖ *Preparation of navel biosorbent from following agricultural byproducts-*

- Sugarcane Bagasse (SB)
- Rice Husk (RH)
- Corn Cob (CC)

❖ *Characterization of the bio-sorbents-*

✓ Field-Emission Scanning Electron Microscopy (FE-SEM)

✓ Fourier-Transform Infra-Red Spectroscopy (FTIR)

✓ X-Ray Diffraction Technique (XRD)

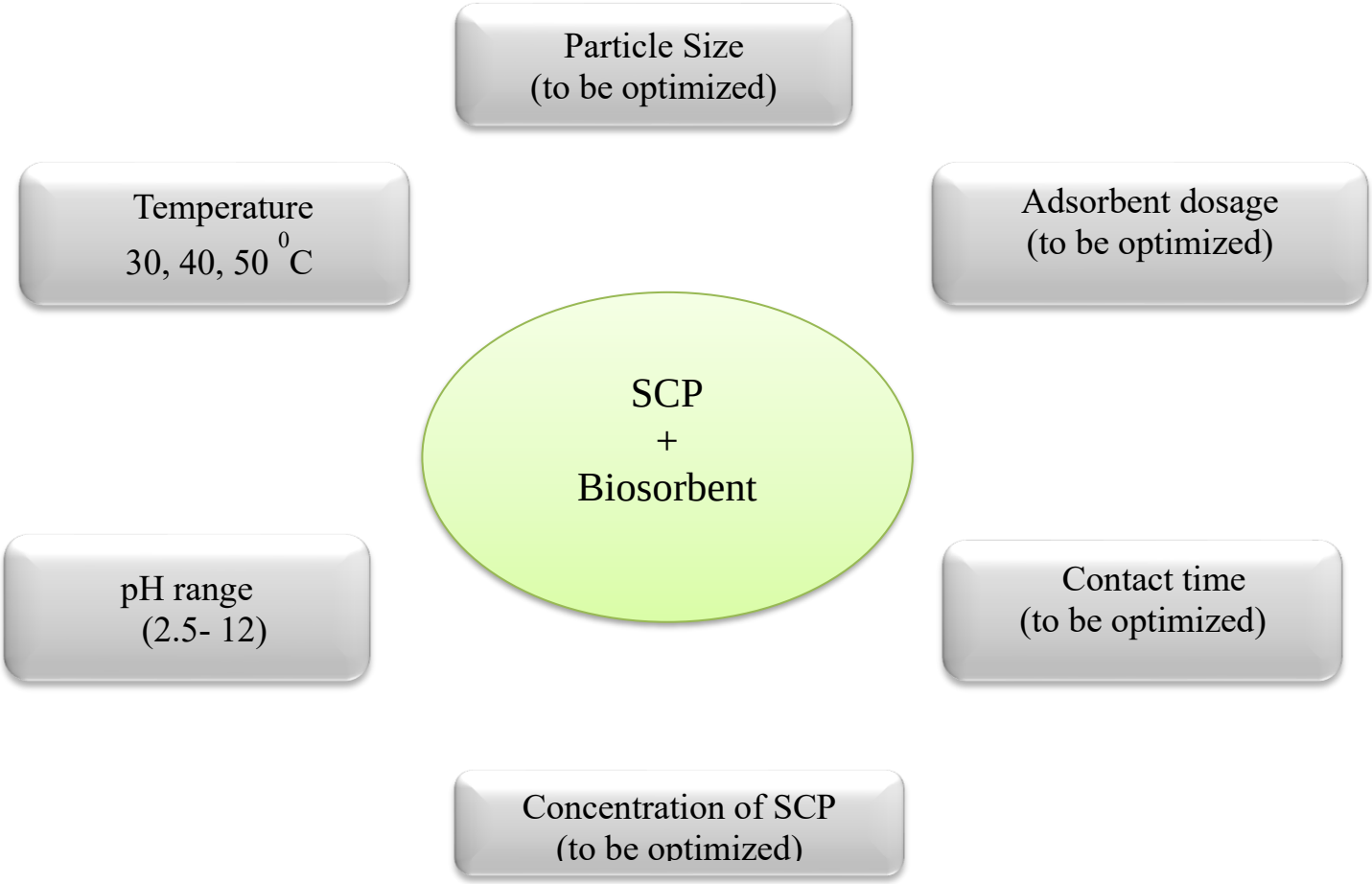
✓ Brunauer-Emmett-Teller (BET) Analysis

✓ Thermo Gravimetric Analysis (TGA)

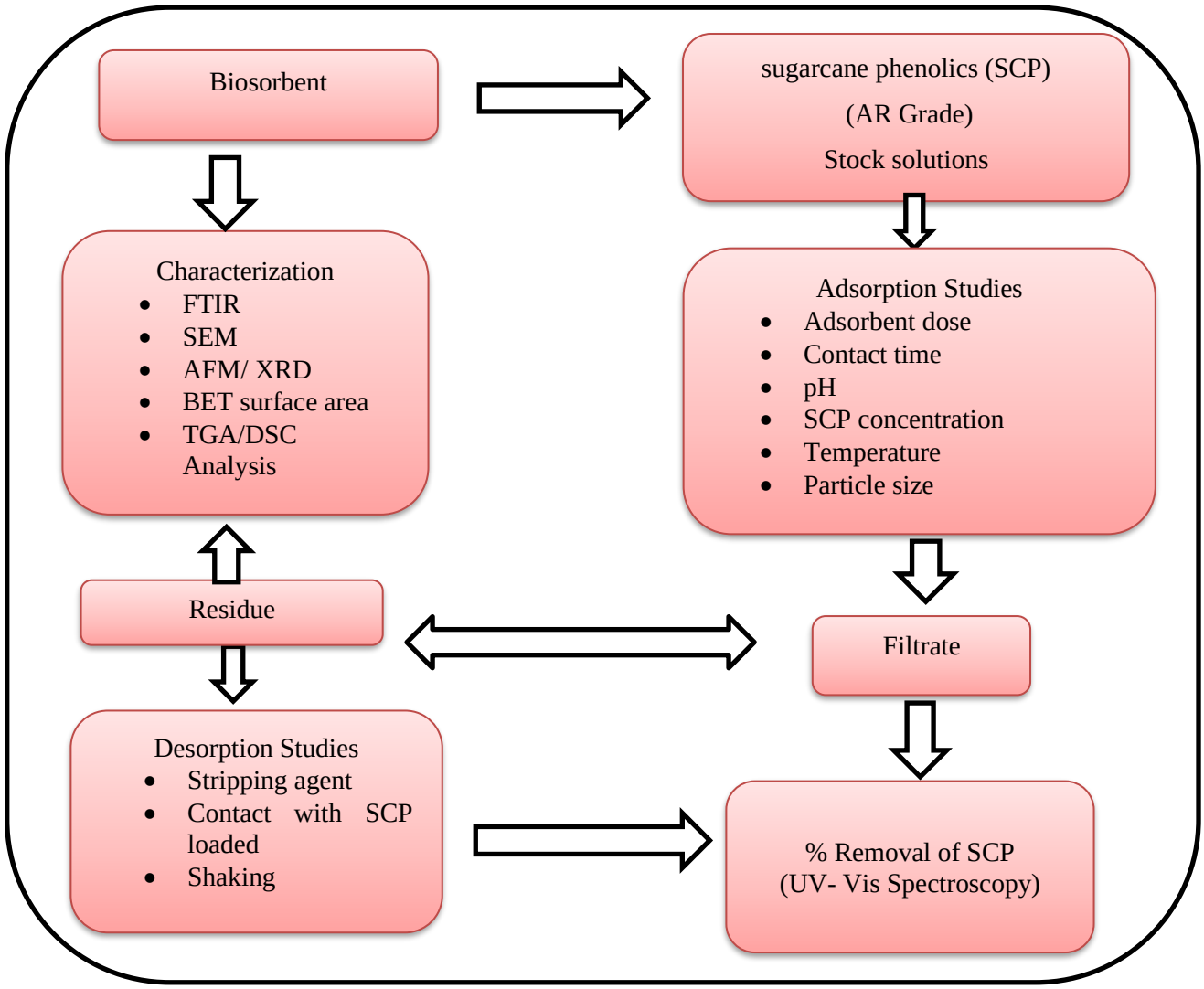
- ✓ Differential Scanning Calorimetry (DSC)
- ✓ Atomic Force Microscopy (AFM)
- ❖ *Study of the following parameters on adsorption in the presence and absence of Ultrasound (US)* -
- ✓ Effect of pH
- ✓ Effect of contact time
- ✓ Effect of temperature
- ✓ Effect of adsorbent dosage
- ✓ Effect of particle size
- ✓ Effect of initial concentration of sugarcane phenolic acid.

EXPERIMENTAL CONDITIONS FOR ADSORPTION

❖ Adsorption efficacy at laboratory scale using standard practices would be carried out in batch experiments under various experimental conditions in the presence and absence of Ultrasound (US)



EVALUATION OF SORPTION AND REGENERATION EFFICIENCY



Methodology used for Sugar Solution Colour Measurement

Analytical potential of the biosorbent is assessed in batch mode and adsorption experiments will be performed. "ICUMSA" (International Commission for Uniform Methods of Sugar Analysis) is a worldwide body which brings together the activities of the National Committees for Sugar Analysis in more than thirty-member countries. An ICUMSA rating is an international unit for expressing the purity of the sugar in solution and is directly related to the colour of the sugar. The industry standard sugar solution colour measurement is the (ICUMSA) colour method [35].

The absorbance of the test solutions will be recorded using UV double beam spectrophotometer. The colours of the sample will be determined using the ICUMSA protocol. ICUMSA colour is an indication of overall colour of sugar solution. The colour will be calculated as follows:

$$\text{I.U.} = \frac{Abs \times 1000}{b \times C}$$

b = cell length (cm)

C = Sugar concentration (gm/ml)

ICUMSA colour (I.U.) is an indication of the overall colour of the sugar solution.

The Colour Removal (CR%) is expressed as follows:

$$CR\% = \frac{I.U_i - I.U_f}{I.U_i} \times 100$$

$I.U_i$ = Initial ICUMSA colour

$I.U_f$ = Final ICUMSA colour

Proposed adsorption mechanism

The proposed adsorption mechanism is based on the charge-transfer mechanism and inter ionic attractions. It is suggested that the phenolic compounds present in solution adsorb on activated sugarcane bagasse ash, rice husk ash and corn cob ash surface by a donor-acceptor complex mechanism involving the surface oxygen of the biosorbent acting as electron donor and aromatic ring of the phenolic acid as the acceptor (figure 2).

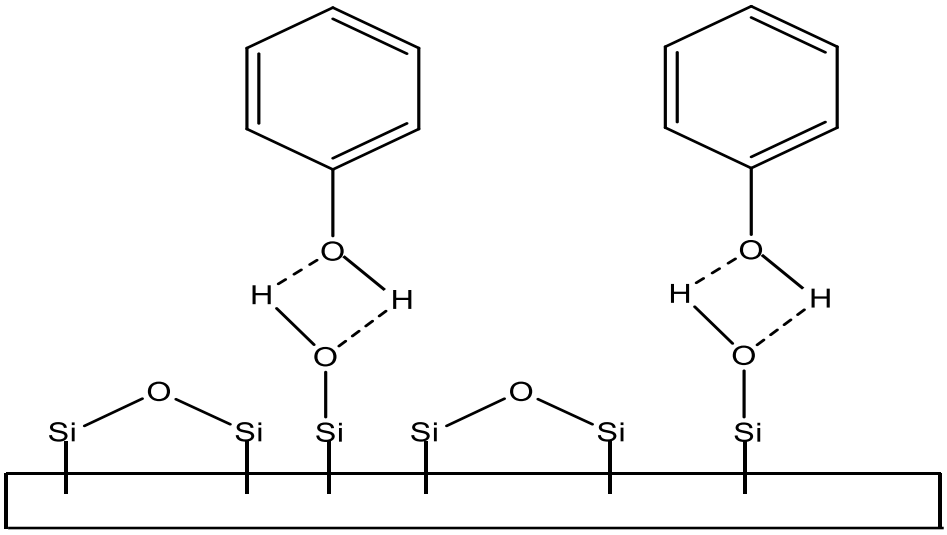


Figure 2. Schematic diagram for the proposed adsorption mechanism of the phenol molecules onto the surface of green biosorbent, through hydrogen bonds between -OH group of the phenol and the silanol groups of the green biosorbent surface. [50]

SIGNIFICANCE OF THE WORK

The research findings may be used for the removal of sugarcane phenolics (sugarcane phenolic acid) or raw sugar colour (dark brown) from aqueous media by green routes. The proposed research work has some important implications in the sugar refining process: -

1. It is aimed to obtain the fundamental data on adsorption of sugar colour from aqueous sugar solution.
2. The obtained results could be applied in sugar industries in various separation processes in addition to removal of sugarcane phenolic acid or sugar colour.
3. The present work is proposed to be undertaken to explore the feasibility of new adsorbents to uptake of sugar colour.
4. The present project may to offer very cheap, efficient and useful adsorbents for effective removal of sugar colour from aqueous sugar solution.
5. To develop new technical process for sugar industries which is beneficial for process control and significant energy saving.

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